NATIONAL BUREAU OF STANDARDS REPORT

9360

Progress Report on Laboratory Tests on 13 West German Dental Silicate Cements by Methods Outlined in Fédération Dentaire Internationale Specification No. 5

U.S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

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Laboratory Tests on 13 West German Dental Silicate Cements by Methods Outlined in Fédération Dentaire International Specification No. 5

Abstract

West German silicate cements were surveyed for compliance with Fédération Dentaire Internationale Specification No. 5. Of the thirteen cements available, eight complied with all requirements for physical and chemical properties. The compressive strengths of the other five cements were below the specification limit. Two of these also had high solubility and one had a longer setting time than is permitted. None of the cements complied with all requirements for packaging, marking and instructions.

1. Introduction

In the USA silicate cements have been tested according to the American Dental Association Specification No. 9 for Dental Silicate Cement since 1938 [8]. The FDI adopted a modification of ADA Specification No. 9 as FDI Specification No. 5 in 1961 [6] and amendments in 1962 [7].

Specifications, established for dental materials, include tests for the most critical physical and chemical properties. For example, for the silicate cements it is of great importance to investigate the solubility and compressive strength. On the other hand when two or more properties are closely related tests may be included for only one of the properties. For example, though the compressive strength of amalgam is important for this material it is not required because most alloys have a satisfactory strength and if they do not it will be detected in another test like the flow test.

It would be very desirable to have well controlled clinical tests for characterized dental materials, but unfortunately most clinical tests are not easily conducted. They are time-consuming and the conditions are difficult to reproduce. It is much easier and sometimes more suitable to depend on laboratory testing. In any event laboratory tests are necessary for a comprehensive evaluation of a dental material. The ADA and FDI silicate cement specifications [1,6,7] include such laboratory tests and give limiting values for certain pertinent physical and chemical properties. The tests can be run in any laboratory and deliver reproducible values. The specifications--especially those adopted by the FDI--are internationally approved and give guiding principles on how to test a certain material. This is an advantage to all other investigations like those recently done by Eichner and co-workers [4,5].

The specifications are established on a foundation of wide clinical experience and laboratory research. All the current FDI specifications have their origin in ADA specifications, and most of the ADA specifications have been in use for decades so they have proved their value. To illustrate how carefully a new ADA specification is set up, when it is found necessary to have a specification for a new material or to change a specification, a committee will collect all obtainable information on the desirable characteristics and the shortcomings of such materials. After a first draft, tests are carried out at various institutions such as the National Bureau of Standards, dental schools, the manufacturers and other interested parties. At this point the manufacturer can give his comments and influence, to some extent, the formulation of the requirements and tests included in the

specifications. A reviewed draft will be submitted and distributed which might be discussed, changed again and finally will be approved by the Specifications Committee and the Council on Dental Research of the ADA. [1] In the end, there is a proved specification which makes it possible to test a material in an appropriate way.

The "Guide to Dental Materials" [1] includes not only the available specifications but also a list of certified materials which have been found to comply with the specifications. By using this list the dentist can select a satisfactory material and does not have to depend on the more or less significant advertisements of the manufacturer. He is also protected against an unproved or unsatisfactory material [3]. The general objective is to exclude poor materials and to offer a selection of good ones.

2. Method

These investigations were done to show which German silicate cements would comply with FDI Specification No. 5 [6,7]. It is contemplated that a certification program will be established in Germany.

All German silicate cements available on the market were procured from distributors. The 13 products listed in Table 1 were tested. The test samples consisted of 6 bottles each instead of the 18 as required by the specification. This was the only exception in the testing procedure.

In testing silicate cements it is very important to make sure that all tests are done at a standard temperature and humidity [8]. In accordance with the specification, the tests were run in a constant temperature room at 23.0 ± 2.0°C and at a relative humidity of 50 ± 10% (4.3.1)*. All tests in which a product failed were repeated.

3. Results

The first examinations were done by inspection and by measuring the weight of the powder and the volume of the liquid. Several discrepancies were found (Table 2).

3.1 Inspection

3.1.1 The liquids were clear, none of them showed any cloudiness. But one (C) had sediment on the bottom of the bottles (3.2.1).

3.1.2 The powders (3.3) were free of extraneous material and the pigment was uniformly dispersed.

3.1.3 The instructions (3.8 and 5.2) were complete for only four products. In the others many discrepancies were noted: For example the temperature of the slab was not given more exactly than just "cool". The powder-liquid ratios were not specific, instead such phrases as "own experience" or "like" other silicate cements" were used. Usually there were detailed instructions for the rate of the powder incorporation, but, exact figures for the time of mixing were not given in four cases (Table 2). A statement for the maximum working time was found for five products only. This statement was required in the 2nd edition of the ADA specification in 1964.

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* These figures in parenthesis refer to sections of FDI Specification No. 5 [6,7].

3.1.4 Leakage was noticed in one case (5.1)*. Perhaps the screw cap was not tightened properly. The effect was not only a loss in volume but a discoloration caused by the red cap (product K).

3.1.5 Lot numbers (5.3) were present in nine cases for the powders and in eight for the liquids (Table 2). For the other silicate cements it is not possible to refer to a certain batch.

The date (5.3.2) was not obviously indicated on the package in any case. Unless the lot numbers are present one never knows if one is using an old or perhaps an improved batch. The requirement for marking the amount of powder and liquid on the container (5.3.3) was generally not complied with. In four cases figures were given for the powder and in three cases for the liquid (Table 2). This is a much more satisfactory procedure than selling an unspecified amount. Nevertheless, it must be mentioned that the liquid was short in two out of those three cases in which the volume was marked on the bottle.

3.2 Physical and chemical tests.

More important than the inspection tests are the physical and chemical tests. Investigating the compressive strengths, opacity, solubility and arsenic content one obtains knowledge about the properties which are significant for the silicate cements. All tests depend very much on the powder-liquid ratio used for the mix. As a general rule, it is considered that the more powder one can get into a certain amount of liquid, the faster is the setting time, the higher is the compressive strength and the lower is the solubility [8]. For this reason and in order to get comparable data the standard consistency test method was established. It provides that varying amounts of powder are mixed with a certain volume of liquid (0.4 ml) until a mix with the required consistency is obtained. The standard testing consistency is defined as that which produces a disk 25 ± 1 mm in diameter (4.3.2; Figure 1) when a load of 2500 g is applied to 0.5 ml of mixed cement for 10 minutes.

3.2.1 Having determined the testing consistency one can calculate if the bottles of the liquid contained the required amount. The liquid must be 20% in excess of that necessary to combine with the powder to make a mix of standard consistency (3.2.2). With the exception of one product (E) all others comply with this requiremrnt (Table 2).

3.2.2 The setting time (Figure 2; 4.3.3) is determined as the time elapsed from the starting of the mix to the time when a standard Gillmore needle (454 g) fails to make a perceptible circle on the surface of a specimen. The established value in the specification is between 3 and 8 minutes. Generally all german silicate cements have a longer setting time than the American ones. One German silicate cement (E) fails because of its long setting time. It would be advisable to reduce this long setting time because it must be presumed that no average dentist will keep the cellophane strip in position for such a long time.

3.2.3 The compressive strength (4.3.4) is defined as the load per square centimeter necessary to break the specimen. According to the established value of a minimum compressive strength of 1700 kg, eight products passed and five failed (Figure 3; Table 3). After storage in distilled water for one day product E showed many cracks from the surface deep into the center of the specimens.

3.2.4 Because of the use of silicate cement as an anterior filling material its optical properties are of interest. There is no specification for the shades, however there exists a test for opacity. The opacity is based on the reflectance from the specimen (1 mm thick) when it is backed by a black background divided by the reflectance when it is backed by a white background. The opacity is measured by comparison with opal glass standards having known values. Opacity does not seem to be a problem,

* These figures in parenthesis refer to the sections of FDI Specification No. 5 [6,7].

because all silicate cements passed this test (Figure 4; Table 3).

3.2.5 The advantages of lifelike color and translucency of silicate cements are counterbalanced by the disadvantage of high solubility. The specifications give a limitation up to 1% by weight for specimens stored for one day in distilled water (4.3.6)*. Eleven of the products passed (Figure 6; Table 3). The two failing are far off the limit (Figure 6). Cement E has an almost five times higher and cement F more than two times higher solubility than the maximum permitted value of 1%. The surfaces of both cements were covered with blisters and crystals after one day storage in distilled water (Figure 5). Not only the specimens for the compressive strength but also those for the solubility of product E cracked.

3.2.6 The need for the arsenic test (4.5.7) is somewhat questionable with modern silicate cements. While in former times a higher arsenic impurity of the cements may have affected the pulp, nowadays the amount of water-soluble arsenic is always so low that this test is only continued to prove the low content. No product failed this test. All had less than 0.0002% arsenic.

4. Discussion

4.1 Testing consistency - The standard testing consistency in the specification is based upon the average consistency used by dentists. Generally it is not the optimum consistency that should be used. The optimum consistency should be specified by the manufacturer in his mixing directions. A rational comparison of the physical properties of silicate cements cannot be made unless they are mixed at a fixed or standard consistency. No valid statement can be made regarding compliance with the specification if the cements have not been mixed at the specified standard consistency. Specifications [2,11] which require mixing at the manufacturer's recommended consistency may admit cements which will not have good properties when used as the average dentist uses them if the manufacturer's recommended consistency differs greatly from that used by the average dentist.

4.2 Instructions - The instructions should carry accurate and adequate statements for the temperature of the slab, the powder-liquid ratio, the rate of powder incorporation, the time of mixing and the maximum working time. Because of the differences between the directions for the practical use in the dental office and those required by the specifications for the standard consistency the manufacturers may use the ingenious way, as in the instructions of product G, of giving an extra item "for scientific investigations" with the necessary statements. For an actual filling no dentist will start weighing the amount of powder and measuring the liquid with a graduated syringe. To incorporate as much powder as possible and still get a workable mixture, cooling of the slab is required.

4.3 Compressive strength - Establishing certain limits for physical and chemical tests is difficult for dental materials. The minimum value for compressive strength in the specification is based upon the strength of the available cements. It is quite certain that a product with cracks after 24 hours going from the surface deep into the specimens, as with product E, and having such a low compressive strength (less than 400 kg/cm^2) cannot be recommended [1,3].

4.4 Solubility - Cracks occurred also on the solubility specimens of cement E. The blisters and the granulation on the surfaces of the products E and F were apparently caused by crystal growth (Figure 5). This indicates that the solubilities were so high that these two silicate cements cannot be recommended [1,3]. One should be very strict with the given limit of 1% solubility, because the solubility is the worst fault of the silicate cements.

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* These figures in parenthesis refer to the sections of FDI Specification No. 5 [6,7].

There has been and there is still much discussion about using distilled water as the solvent for the solubility test. Acids and especially the change between a basic and acid solvent cause a much higher solubility [5,9,10]. Since there is no final resolution and the conditions for solubility differ widely in the mouth, one should use the most common and generally used inorganic solvent: distilled water, as no satisfactory substitute has been presented.

4.5 General Discussion - Generally it cannot be expected that dental materials which never before have been subjected to specification testing will comply in every detail. Even though the dentist may not read the instructions that is no reason for giving only an advertisement for a nice bottle as with product K and leaving the way of mixing and using to the dentist. In all instances each product was lacking some details in the instructions or marking. This can be changed easily by adding the required statements to the instructions or printing the net weight or net volume on the label. More severe than these formalities are deficiencies in physical and chemical properties. It was supposed that the greatest trouble would be the solubility. But only two products failed in this test. It seems to be much more difficult to get an adequate compressive strength.

There were generally no exceptions to the requirements for purity of the liquids and powders, to the setting time and opacity.

All these cases where the cements did not comply with the specifications show that it is of extraordinary importance to use the FDI specifications and to establish a specification program in Germany in order to recommend only the best materials to the dental profession. Once the manufacturers become accustomed to their products being tested by an independent institution, lacking details and properties will be eliminated or the material discontinued and products will improve further.

5. Conclusion and Summary

Thirteen German silicate cements (Table 1) were tested according to FDI Specification No. 5. The instructions, packing and marking of all products showed a number of inadequacies varying from product to product (Table 2). Eight products passed, five failed in the physical and chemical properties required in the specification (Table 3). One product (E) had an extremely low compressive strength (Figure 3), two (E and F) had extensive solubility (Figure 6).

6. Bibliography

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Table 1

Materials Investigated

Manufacturer	Brand
Bayér A. G.	BS 58 with liquid "normal"
" " "	BS 58 with liquid "quick"
De Trey GmbH	Super Syntrex
Drala GmbH	Translucin
Harvard Gesellschraft	Crystone
Jota GmbH	Omnifil
Mamorlaboratorium	Pleveth
" " "	Faser Pleveth
" " "	Mamorith
" " "	Super Mamorith
Süd-Dental	Boston
Zahn-Porzellan A. G.	Terralux
Zhanel	Zhanelka

This arrangement does not correspond to the order of the letters A, B, C, through N for the different products.

Table 2

Compliance with Inspection Requirements

(+ = complies, (+) = incomplete, - = fails)

product	liquid			powder	Instructions						leak-proof containers		marking			
													powder		liquid	
	clear	sediment	20% excess			a) temperature of slab	b) powder-liquid ratio	c) rate of powder incorporation	d) time of mixing	e) maximum working time	powder	liquid	lot no.	weight marked	lot no.	volume marked
A	+	+	+	+	+	(+)	-	+	+	-	+	+	+	-	+	-
B	+	+	+	+	+	(+)	-	+	+	-	+	+	+	-	+	-
C	+	-	+	+	+	-	(+)	(+)	-	-	+	+	+	-	+	-
D	+	+	+	+	+	(+)	-	+	+	-	+	+	+	-	(+)	-
E	+	+	-	+	+	+	-	+	-	-	+	+	-	+	-	-
F	+	+	+	+	+	+	+	+	+	+	+	+	-	-	-	-
G	+	+	+	+	+	+	+	+	+	-	+	+	+	+	+	+
H	+	+	+	+	+	+	+	+	+	+	+	+	(+)	+	+	+*
I	+	+	+	+	+	+	+	+	+	+	+	+	(+)	+	+	+*
K	+	+	+	+	+	-	-	-	-	-	+	-	+	-	-	-
L	+	+	+	+	+	+	+	+	+	-	+	+	-	-	-	-
M	+	+	+	+	+	+	-	+	-	+	+	+	-	-	-	-
N	+	+	+	+	+	+	+	+	+	+	+	+	(+)	-	(+)	-

* The actual volume was less than marked.

Table 3

Physical and Chemical Properties*

	Standard consistency $\frac{g}{0.4\ ml}$	Time of setting	Compressive strength at 24 hours	Opacity at 24 hours $C_{o.7o}$	Solubility
Limits Min.:	-	3 min.	1700 $\frac{kg}{cm^2}$	0.35	-
Max.:	-	8 min.	-	0.55	1%
Product	g	min.	$\frac{kg}{cm^2}$		Percentage by weight
A	1.50	8	1810	0.40	0.7
B	1.50	8	1980	0.35	0.6
C	1.40	7	1610	0.40	0.7
D	1.45	7	1870	0.35	0.7
E	1.05	10	380	0.50	4.8
F	1.05	6	1570	0.45	2.2
G	1.55	5	2180	0.35	0.5
H	1.50	8	1780	0.35	0.7
I	1.45	7	1890	0.35	0.7
K	1.40	5	1730	0.35	0.6
L	1.25	6	2070	0.40	0.8
M	1.45	6	1450	0.35	0.6
N	1.50	5	1480	0.40	0.8

* Underlined values do not comply with the specification. All the cements had less than 2 parts per million of water-soluble arsenic as required.

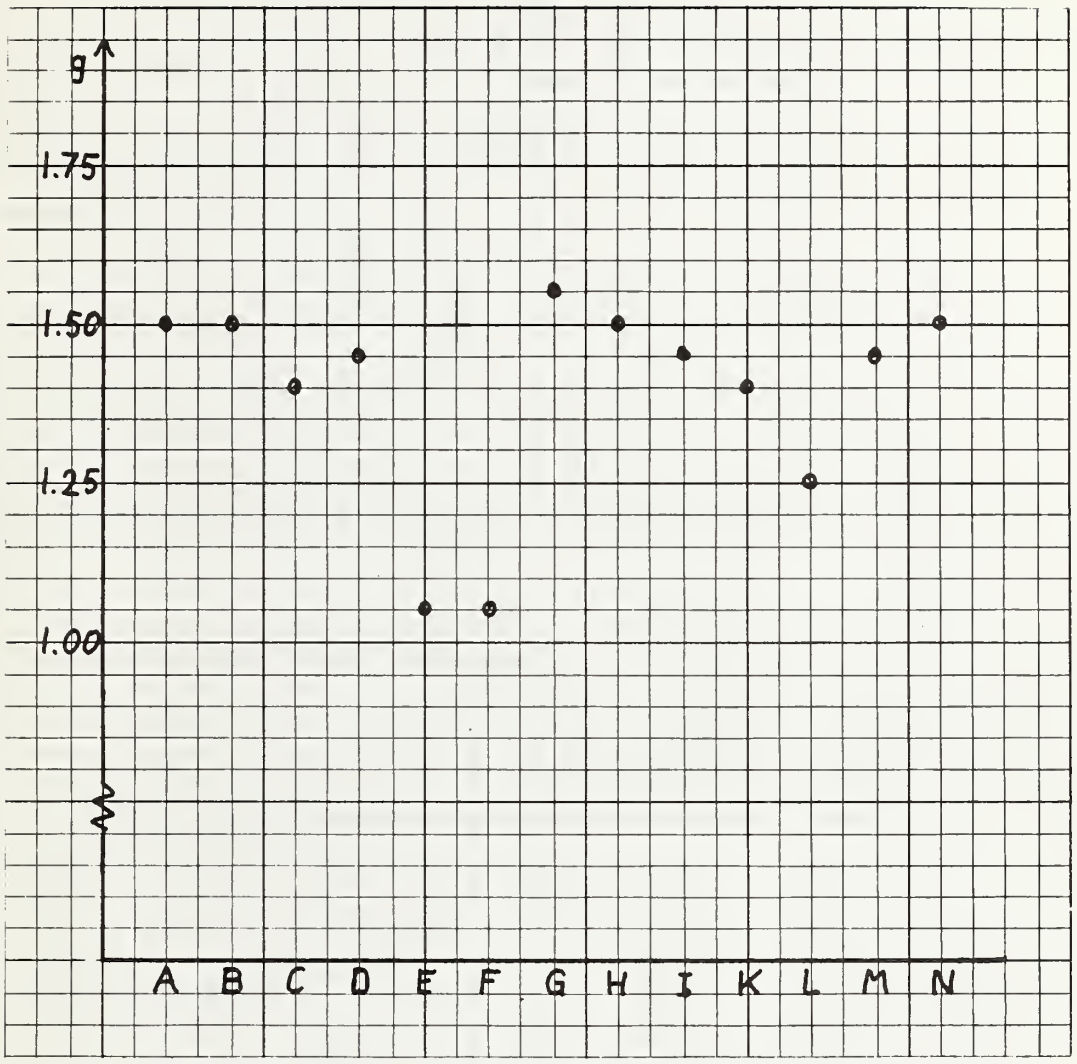

Figure 1. Testing consistency. Grams of powder in 0.4 ml liquid, necessary to produce a disk of 25 ± 1 mm in diameter.

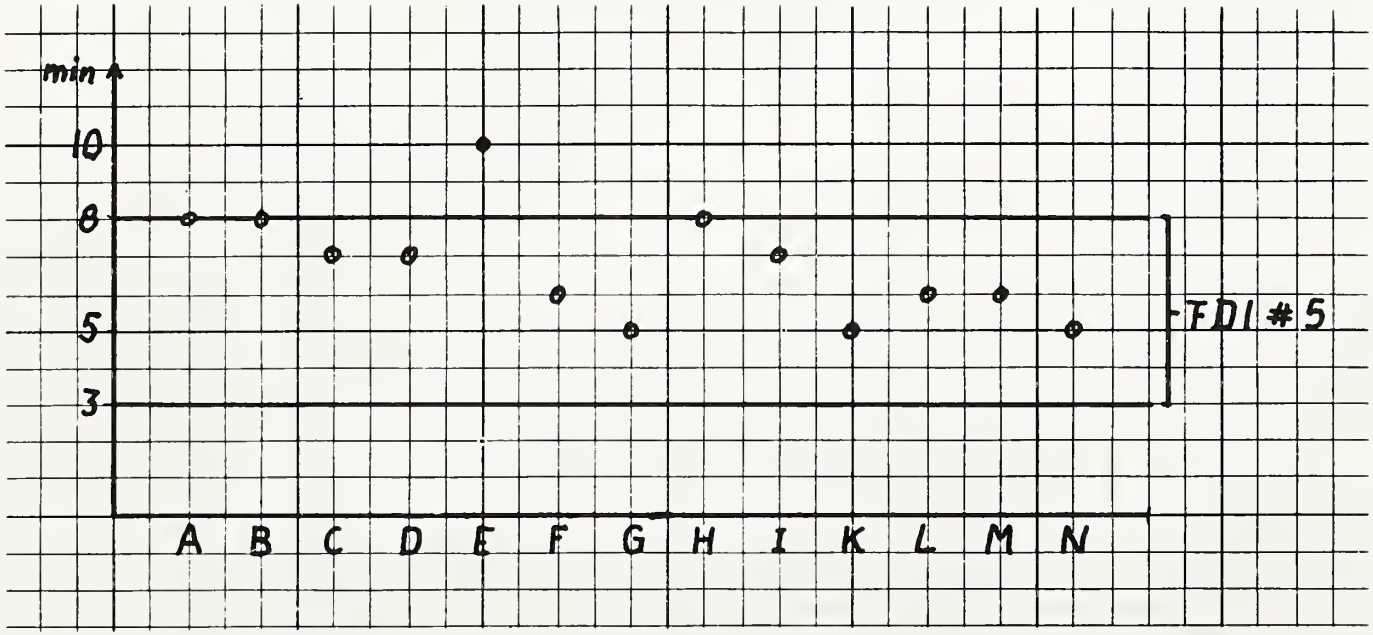

Figure 2. Setting time (limits: 3 - 8 min).

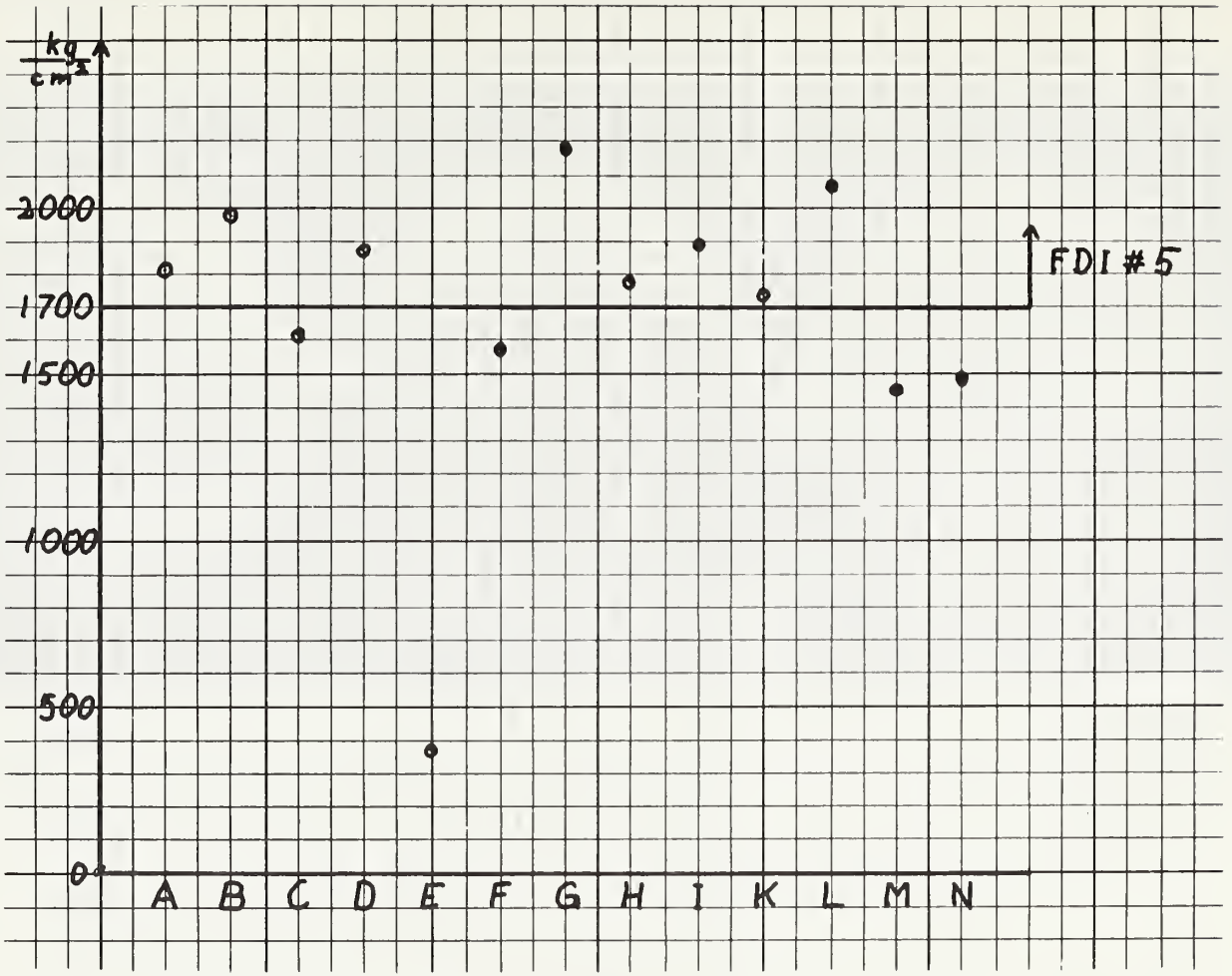

Figure 3. Compressive strength (limit: min. 1700 $\frac{kg}{cm^2}$).

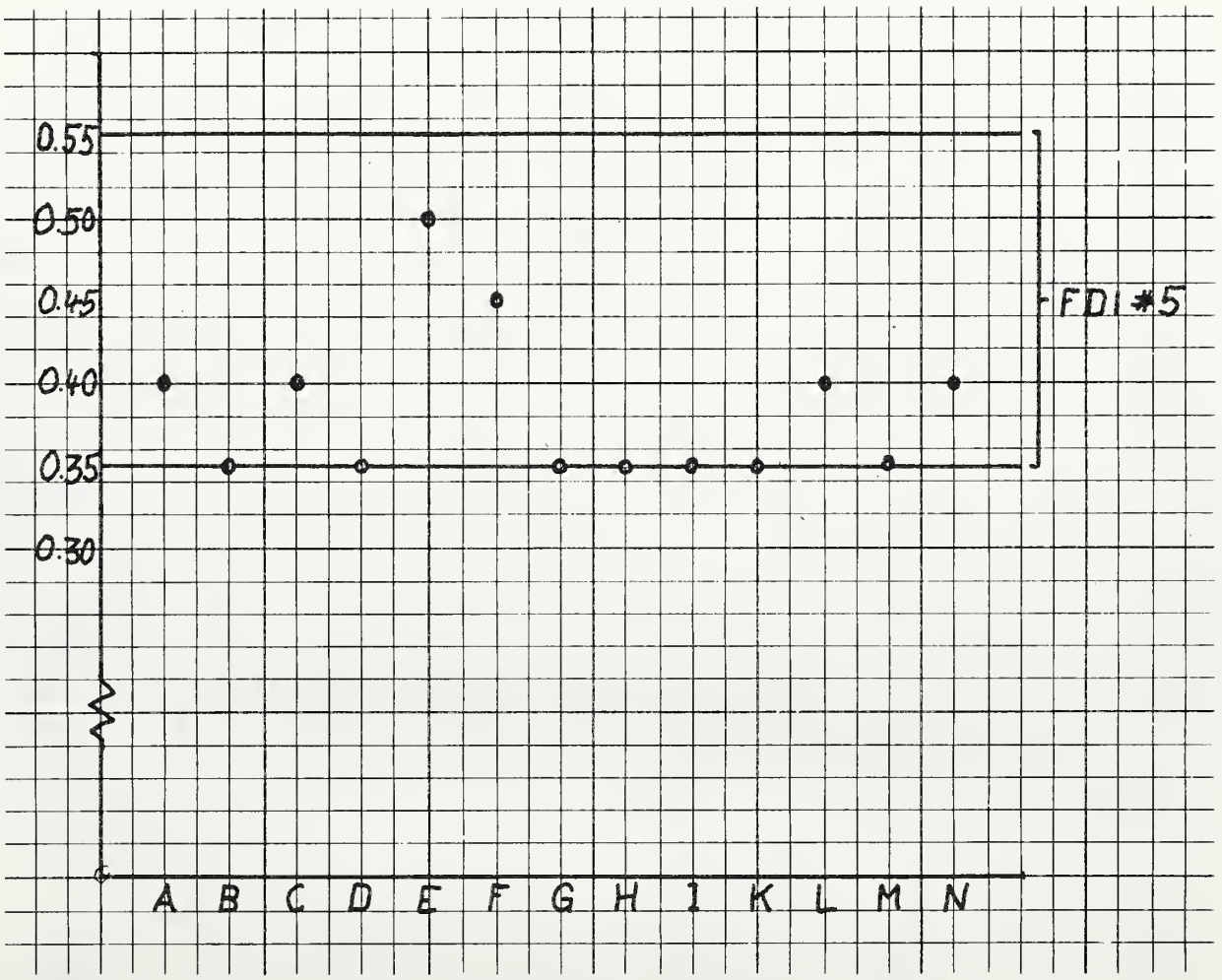

Figure 4. Opacity (limits: 0.35 - 0.55 with a $C_{o.7o}$ backing).

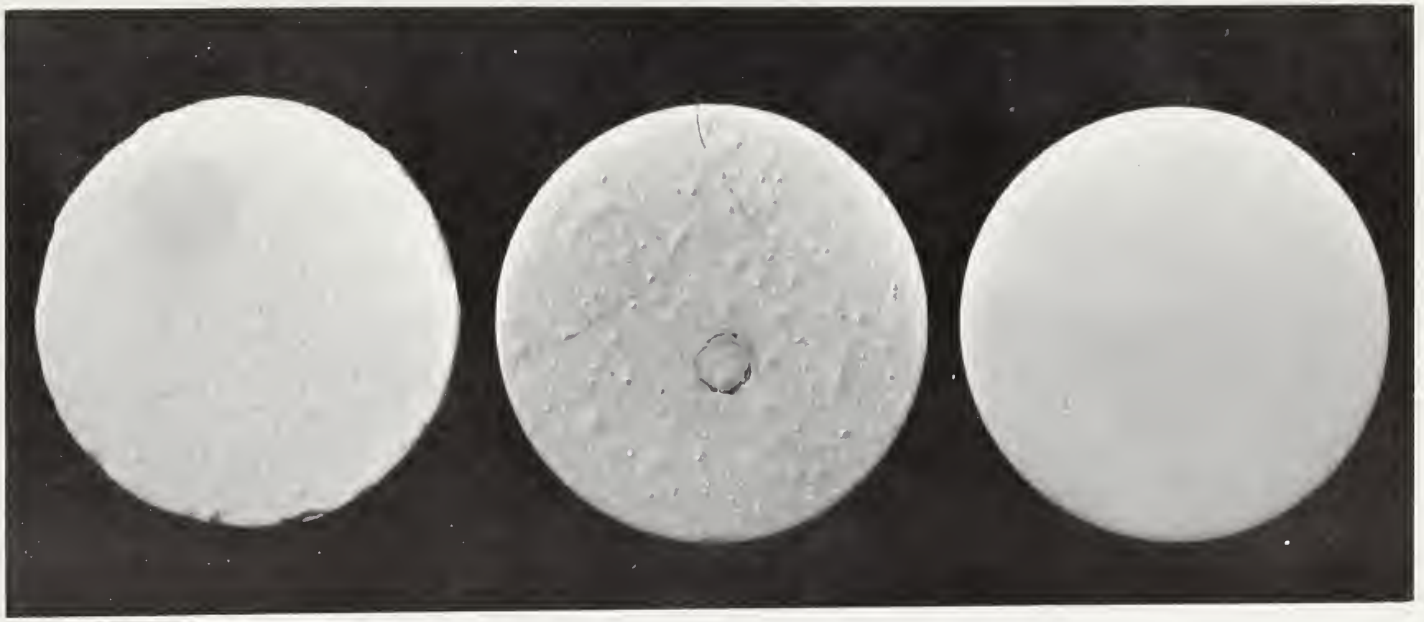

Figure 5. Solubility specimens (20 mm diameter) after 23 hours in distilled water.

Left: Product E, crystal growth, bubbles, broken edges.

Center: Product F, crystal growth and bubbles, in the middle indentation caused by the platinum wire.

Right: Product H, desirable smooth surface.

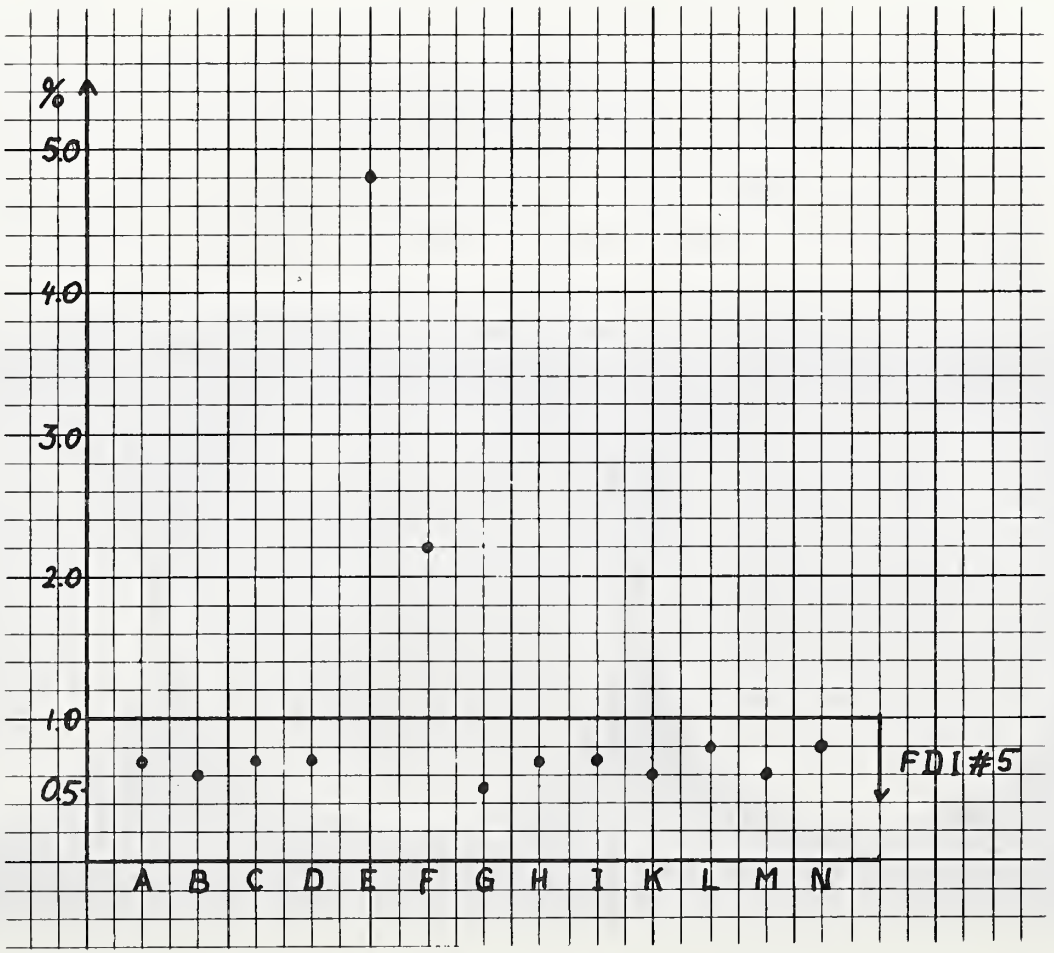

Figure 6. Solubility and disintegration (limit: max. 1%).


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